# Effect of pH on the Phase Equilibrium of a Ternary Mixture of Neopentyl Glycol, Salt, and Water

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The ternary two-liquid phase system consisting of neopentyl glycol, sodium formate, and water was studied at pH values of 3, 4, 5, and 6. This study examined the effect of pH on phase diagrams, focusing on the length and slope of the tie-lines between the two conjugated phases. The results showed that the two-phase region expanded, and both the length and slope of the tie-lines increased with rising pH values. The phase behavior was analyzed using the Bancroft, Othmer-Tobias, and Hand equations, the measured data showing acceptable consistency, and the Bancroft equation providing the best fit. The plait points at different pH values were estimated through extrapolation, and empirical equations were used to fit the data and gave good results. The fitted curves matched the data well.

*Keywords*

aqueous two-phase systems, neopentyl glycol, phase equilibria, pH value, sodium formate

## Introduction

Neopentyl glycol is a colorless, odorless crystalline solid, that is easily soluble in water, alcohol, ketone, ether, hot aromatic solvents, while it is insoluble in aliphatic hydrocarbons and cycloalkanes. Its molecular structure, characterized by two symmetrical primary hydroxyl groups and a lack of α-hydrogen atoms on the central carbon atom, enables it to participate in a unique set of chemical reactions such as esterification, etherification, condensation and oxidation. Neopentyl glycol molecules and its derivatives exhibit high thermal and chemical stability, making it a valuable raw material in a wide range of applications. It is primarily used in the production of various new resins, plasticizers, synthetic lubricants, surfactants, insulating materials, pharmaceuticals, flame retardants, and more[1–3].

Currently, neopentyl glycol is produced via two primary methods: the disproportionation process, and the hydrogenation process. The hydrogenation process, however, requires significant investment, has strict environmental requirements, and results in a costly product. To mitigate these drawbacks, the sodium formate method can used, producing neopentyl glycol at a lower cost. In this method, formaldehyde and isobutylaldehyde are first reacted to form hydroxy-tert-butylaldehyde under specific pH and temperature conditions, which is then converted to neopentyl glycol by excess formaldehyde in strong alkali conditions. Simultaneously, formaldehyde reacts with the alkali and oxidizers to form sodium formate. After reaction, the solution undergoes dehydration by distillation, sodium formate removal, cooling, crystallization, and separation to yield the final product. During this process, a notable quantity of sodium formate is produced, which is considered the primary by-product. The removal and separation of the by-product (sodium formate) directly affect the purity and quality of neopentyl glycol. Therefore, it is crucial to study the relationship between the solubility of neopentyl glycol and sodium formate in aqueous solutions under varying pH and temperature conditions. However, apart from a preliminary study by the authors of this paper on the dissolution process at different temperatures, there has been no report on this ternary system. At the same time, the authors have found that an aqueous solution of neopentyl glycol and sodium formate can form a two-phase system. Liquid-liquid extraction using aqueous two-phase systems is a kind of extraction separation technology, which has gained increased attention in recent years. Specifically, small molecule organic solvent/salt systems are valued for their rapid phase separation, high selectivity, and good extraction yield, leading to increased interest in the development and research of aqueous two-phase systems[4–6].

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Our previous research focused on the dissolution process and liquid-liquid equilibrium of neopentyl glycol, salt and water systems at different temperatures. Building on that foundation, this study measured the phase equilibrium data of the same system at different pH values at 303.15 K. These findings provide a theoretical basis for the separation of neopentyl glycol and sodium formate, and contribute to establishing a thermodynamic model for the aqueous two-phase separation system of neopentyl glycol and salt.

## Experimental

### Materials

Neopentyl glycol (NPG, purity 99 %) was provided by Shanghai Chuangsai Technology Co., Ltd. Sodium formate (HCOONa, AR, 99.5 %) was sourced from Shanghai Aladdin Biochemical Technology Co., Ltd. Both reagents were used without further purification. Hydrochloric acid standard solution was obtained from Guangzhou Hewei Pharmaceutical Technology Co., Ltd. All solutions were prepared with doubly distilled deionized water. For liquid chromatography, ultrapure water from the Hyperpurex purification system was used.

### Experimental procedure

Weighing bottles were used to determine tie-lines and phase equilibrium. The feed samples with at least seven distinct known total compositions were prepared in the bottles, and placed in a thermostatic bath at the desired temperature with a temperature control accuracy of ±0.5 °C (DK-S14, Shanghai Senxin Experimental Instrument Co., Ltd.). A specified amount of aqueous solution of known pH was added, followed by varying proportions of neopentyl glycol and sodium formate. The pH of the aqueous solutions was adjusted using a precision pH meter (model: PHSJ-4F, Shanghai Lei Magnetic Instrument Factory). The combinations were mixed and allowed to dissolve for two hours at the specified temperature and pH. To ensure sufficient separation of phases, the solutions were left to stand for twelve hours. When the solutions reached phase equilibrium and separated into two phases, the samples were extracted from the top and bottom phases of the equilibrium systems using syringes[7].

### Analytical method

The components in the samples were analyzed for mass concentration using liquid chromatography (Dionex, U-3000) after weighing and dilution. The analytical conditions for liquid chromatography were as follows: a chromatography column (C18: 150 mm×4.6 mm×5 μm), column temperature and detector temperature set at 50 °C. Ultrapure water was used as the mobile phase with a flow rate of 1.5 mL min$^{-1}$. A differential refractive detector was used, and the injection volume was 50 μL [7].

## Results and discussion

### Phase equilibrium

Data were collected for the liquid-liquid equilibrium compositions at four pH values (3.00, 4.00, 5.00, 6.00) at 303.15 K to examine the impact of pH on the phase diagrams in the ternary system of neopentyl glycol, sodium formate, and water. Mass fraction was used to express all compositions, and the data collected are presented in Table 1.

As shown in Table 1, the upper phase appears to contain a higher concentration of neopentyl glycol, whereas the bottom phase seems to be richer in salt (sodium formate). Additionally, Figs. 1 and 2 depict the phase diagrams of the system at various pH values. Table 1 and Figs. 1 and 2 indicate that sodium formate addition resulted in the separation of the neopentyl glycol water mixture.

This liquid-liquid two-phase separation can be attributed to the salting-out effect, driven by the differing hydration rates and the actions of the two components, upon addition of sodium formate salt. The effects of pH (3.00, 4.00, 5.00, and 6.00) on the binodal data for the neopentyl glycol + sodium formate + water systems are presented in Fig. 2. The binodal curve delineated two regions: the homogeneous phase region and the heterogeneous (two-phase) region. It is evident from Fig. 2 that the mixtures with total initial compositions below the binodal lines formed a homogeneous single-phase
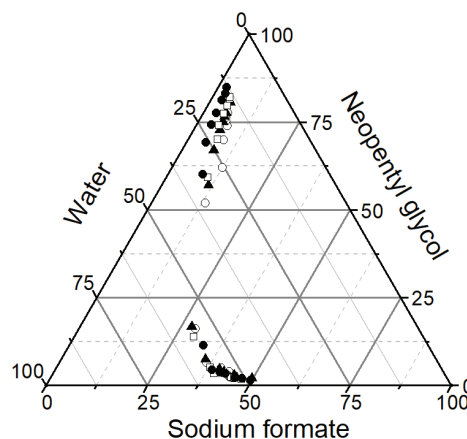


F i g . 1 – *Equilibrium phase diagram of the neopentyl glycol + sodium formate + water two-phase system at 303.15 K and pH values of 3.00 (○), 4.00 (▲), 5.00 (□), and 6.00 (●)*

Table 1 – *Phase equilibrium binodal data, tie-line lengths, TLL, and slope of tie-lines, S, of the neopentyl glycol (1) + sodium formate (2) + water (3) system for different pH values at 303.15 K*

| pH | Top phase | | Bottom phase | | 100 TLL | S |
|---|---|---|---|---|---|---|
| | $100w_{(1)}$ | $100w_{(2)}$ | $100w_{(1)}$ | $100w_{(2)}$ | | |
| 3.00 | 52.00 | 13.28 | 16.16 | 28.91 | 39.10 | –2.29 |
| | 62.05 | 12.56 | 6.55 | 36.38 | 60.40 | –2.33 |
| | 69.96 | 8.97 | 4.94 | 40.80 | 72.39 | –2.04 |
| | 73.87 | 7.91 | 3.90 | 43.30 | 78.41 | –1.98 |
| | 75.33 | 6.82 | 2.68 | 43.77 | 81.51 | –1.97 |
| | 76.28 | 6.39 | 2.38 | 44.35 | 83.08 | –1.95 |
| | 77.64 | 6.08 | 2.06 | 45.11 | 85.06 | –1.94 |
| 4.00 | 57.13 | 11.59 | 16.62 | 27.66 | 43.59 | –2.52 |
| | 66.97 | 7.95 | 7.54 | 35.63 | 65.56 | –2.15 |
| | 72.78 | 6.53 | 4.65 | 40.55 | 76.15 | –2.00 |
| | 75.11 | 6.15 | 3.74 | 42.01 | 79.87 | –1.99 |
| | 76.67 | 5.74 | 2.98 | 44.92 | 83.46 | –1.88 |
| | 77.62 | 5.61 | 2.27 | 45.94 | 85.47 | –1.87 |
| | 80.64 | 5.21 | 1.97 | 49.90 | 90.48 | –1.76 |
| 5.00 | 59.25 | 10.27 | 13.84 | 29.48 | 49.31 | –2.36 |
| | 70.16 | 7.22 | 5.02 | 38.07 | 72.07 | –2.11 |
| | 74.15 | 5.60 | 3.43 | 39.78 | 78.55 | –2.07 |
| | 77.47 | 5.17 | 3.34 | 42.52 | 83.01 | –1.98 |
| | 79.64 | 4.90 | 2.32 | 44.52 | 86.88 | –1.95 |
| | 81.23 | 4.58 | 2.07 | 45.44 | 89.08 | –1.94 |
| | 82.20 | 4.25 | 1.86 | 47.32 | 91.15 | –1.87 |
| 6.00 | 60.11 | 8.68 | 11.38 | 33.26 | 54.58 | –1.98 |
| | 69.23 | 4.87 | 4.42 | 38.77 | 73.14 | –1.91 |
| | 74.21 | 3.82 | 3.69 | 41.16 | 79.79 | –1.89 |
| | 77.65 | 3.25 | 3.32 | 42.66 | 84.13 | –1.89 |
| | 81.16 | 2.89 | 2.12 | 45.54 | 89.81 | –1.85 |
| | 83.20 | 2.66 | 1.89 | 47.48 | 92.84 | –1.81 |
| | 84.89 | 2.23 | 1.41 | 49.82 | 96.09 | –1.75 |



Fig. 2 – *Effect of pH on the phase diagram and experimental fitting curves for the neopentyl glycol + sodium formate + water system at different pH values at 303.15 K*

neopentyl glycol and water decreased with increasing pH value. Compared to salt ions, the molecules of neopentyl glycol exhibited reduced hydration and solubility in water, causing the alcohol molecules to be expelled from the solution, thereby promoting the formation of two distinct phases. Conversely, as the pH decreased, the amount of $H^+$ gradually increased, leading to the protonation of neopentyl glycol. This enhanced the binding affinity between organic alcohol with water molecules, while reducing the binding competitiveness of salt molecules for water, making phase separation more difficult. This underscores the significant influence of pH on the salting-out effect and the ease of phase separation[12–14].

**Tie-lines**

To examine the influence of pH on equilibrium phase compositions of the studied systems, we calculated the tie-line length (TLL) and tie-line slope (S) at various pH values and compositions using Eq. (1) and (2), respectively[15–17].

$$\text{TLL} = \left[ \left( w_{(1)}^{t} - w_{(1)}^{b} \right)^2 + \left( w_{(2)}^{t} - w_{(2)}^{b} \right)^2 \right]^{0.5} \quad (1)$$

$$S = \left( w_{(1)}^{t} - w_{(1)}^{b} \right) / \left( w_{(2)}^{t} - w_{(2)}^{b} \right) \quad (2)$$

Where, $w_{(i)}$ is the mass fraction of the neopentyl glycol, sodium formate, water. Superscripts 't' and 'b' refer to the top and bottom phases, respectively. The final two columns of Table 1 present the calculated values for TLL and S at various compositions and pH values (3.00, 4.00, 5.00, and 6.00). Fig. 3 illustrates the phase diagram with the tie-lines of the two-conjugate phase (through the top and bottom phase points) at pH 4.00. For illustration purposes, Fig. 4 compares the tie-lines obtained from experimental equilibrium phase composition data for pH 3.00 and 6.00.

system, whereas those with compositions above the binodal line separated into two immiscible phases at equilibrium[8–11].

**Effect of pH on phase diagrams**

Fig. 2 shows the effect of pH on the correlated binodal curves of the neopentyl glycol + sodium formate + water system. The results indicate that as the pH of the system increased, the two-phase region expanded. Specifically, the binodal curve shifted downward with increasing pH, leading to broader two-phase region. This may be attributed to the fact that the hydrogen bonding interactions between
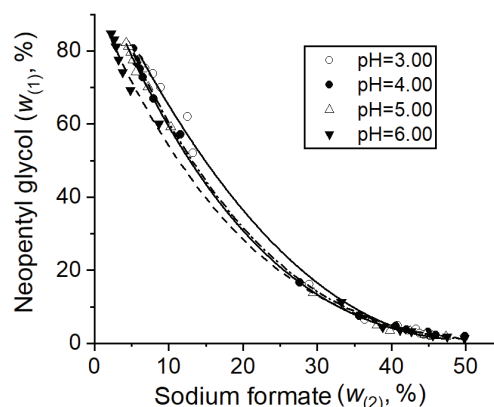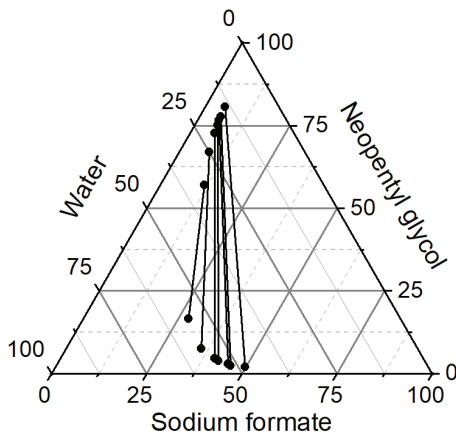
F i g . 3 – *Equilibrium phase diagram and tie-line of the neopentyl glycol + sodium formate + water system at pH= 4.00*
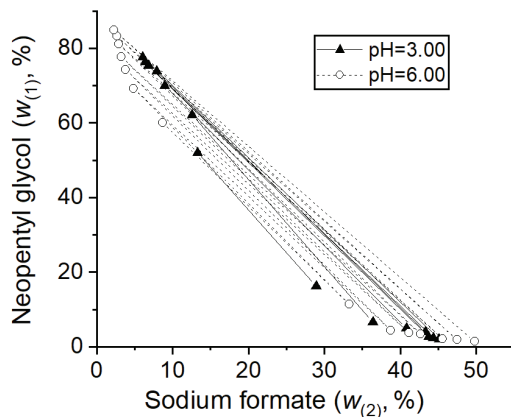


F i g . 4 – *Effect of pH on the equilibrium phase compositions, slope, and tie-line length of the neopentyl glycol + sodium formate + water system*

Table 1 and Fig. 4 demonstrate that the length of the tie-lines increased as the pH of the aqueous medium increased. While the slope values exhibited slight variations among the different tie-lines of a given system, the data indicated that as the pH increased, the value of S also increased. The parameters TLL and S are commonly used as indicators of concentration differences between the upper and lower phase components in liquid-liquid equilibrium. The concentration changes in neopentyl glycol and sodium formate can be attributed to the gradual increase in neopentyl glycol concentration in the rich-phase and its decrease in the poor-phase. Con-

versely, the concentration of sodium formate decreases in the rich-phase and increases in the poor-phase. Notably, the changes in neopentyl glycol concentration were more pronounced than those of sodium formate.

The empirical correlation equations provided by Hand (Eq. (3)), Othmer-Tobias (Eq. (4)) and Bancroft (Eq. (5)) were used to control and ensure the reliability and consistency of data obtained from the tie-line compositions[18–20].

$$\log\left(\frac{w_{(3)}^t}{w_{(1)}^t}\right) = a + b \log\left(\frac{w_{(3)}^b}{w_{(2)}^b}\right) \tag{3}$$

$$\log\left(\frac{1-w_{(1)}^t}{w_{(1)}^t}\right) = c + d \log\left(\frac{1-w_{(2)}^b}{w_{(2)}^b}\right) \tag{4}$$

$$\left(\frac{w_{(3)}^b}{w_{(2)}^b}\right) = e\left(\frac{w_{(3)}^t}{w_{(1)}^t}\right)^f \tag{5}$$

Table 2 presents the fitting parameters for the Hand, Othmer-Tobias, and Bancroft models, represented by $a$, $b$, $c$, $d$, $e$, and $f$, along with their corresponding correlation coefficient values ($R^2$). Based on the correlation coefficient values obtained, it can be concluded that the liquid-liquid equilibrium of the studied aqueous two-phase systems was well-described by these equations, indicating that the data obtained with these models was reliable. Among the models, the Bancroft model provides a better fit compared to the Hand and Othmer-Tobias models.

**Estimation of critical point**

At the critical, or plait, point on the binodal curve, the tie-line length becomes zero as the two liquid phases become identical, making phase separation impossible. The locations of the plait points' for this system at different pH values were estimated by extrapolating from the auxiliary curves[21]. The auxiliary curve is represented by Eq. (6):

$$w_{(1)} = a_0 + b_0 w_{(2)} \tag{6}$$

where $a_0$ and $b_0$ are the fitting parameters in the auxiliary line, and $w_{(1)}$, $w_{(2)}$ represent the mass concentrations of neopentyl glycol and sodium formate

T a b l e 2 – *Values of parameters in Eq. (3), (4) and (5) for the neopentyl glycol + sodium formate + water system at different pH values*

| pH | a | b | $R^2$ | c | d | $R^2$ | e | f | $R^2$ |
|---|---|---|---|---|---|---|---|---|---|
| 3.00 | −0.8233 | 2.3078 | 0.9947 | −0.6570 | 1.6596 | 0.9813 | 2.2684 | 0.4312 | 0.9957 |
| 4.00 | −0.7462 | 1.5606 | 0.9921 | −0.6272 | 1.2051 | 0.9963 | 2.9446 | 0.6236 | 0.9944 |
| 5.00 | −0.8646 | 2.0325 | 0.9929 | −0.7399 | 1.5667 | 0.9887 | 2.6524 | 0.4891 | 0.9947 |
| 6.00 | −0.8296 | 2.3897 | 0.9888 | −0.7744 | 1.9928 | 0.9922 | 2.1853 | 0.4049 | 0.9917 |

T a b l e  3 – *Values of parameters of the auxiliary curves and the plait points, for the neopentyl glycol + sodium formate + water system at different pH values*

| pH | $a_0$ | $b_0$ | $R^2$ | Plait point ($w_{(2)}$, $w_{(1)}$) |
|---|---|---|---|---|
| 3.00 | 5.1398 | 1.5963 | 0.9951 | (19.8588, 36.8408) |
| 4.00 | 28.4273 | 1.0744 | 0.9841 | (14.9646, 44.5049) |
| 5.00 | 20.1026 | 1.3352 | 0.9917 | (15.8325, 41.2425) |
| 6.00 | 9.7778 | 1.5468 | 0.9776 | (16.7363, 35.6651) |

T a b l e  4 – *Values of parameters in Eq. (7), (8), (9) (10), and (11) for the neopentyl glycol + sodium formate + water system at different pH values*

| pH | $a_1$ | $b_1$ | $c_1$ | $d_1$ | $R^2$ |
|---|---|---|---|---|---|
| 3.00 | 98.0585 | −3.2772 | $-4.1441 \cdot 10^{-5}$ | $5.7661 \cdot 10^{-4}$ | 0.9964 |
| 4.00 | 102.6659 | −4.8903 | 0.0744 | $-3.3592 \cdot 10^{-4}$ | 0.9993 |
| 5.00 | 100.7130 | −4.7536 | 0.0691 | $-2.6989 \cdot 10^{-4}$ | 0.9995 |
| 6.00 | 92.9533 | −4.6171 | 0.0797 | $-4.8401 \cdot 10^{-4}$ | 0.9978 |

| pH | $a_2$ | $b_2$ | $c_2$ | $R^2$ | |
|---|---|---|---|---|---|
| 3.00 | −17.4723 | −125.3668 | 0.9585 | 0.9942 | |
| 4.00 | −6.7752 | −116.0347 | 0.9454 | 0.9985 | |
| 5.00 | −8.4759 | −114.3678 | 0.9475 | 0.9988 | |
| 6.00 | −7.0781 | −102.0047 | 0.9486 | 0.9981 | |

| pH | $a_3$ | $b_3$ | $c_3$ | $R^2$ | |
|---|---|---|---|---|---|
| 3.00 | 4.5167 | −0.0178 | −0.0014 | 0.9972 | |
| 4.00 | 4.6017 | −0.0401 | $-8.8097 \cdot 10^{-4}$ | 0.9993 | |
| 5.00 | 4.5904 | −0.0403 | $-9.5482 \cdot 10^{-4}$ | 0.9994 | |
| 6.00 | 4.5336 | −0.0491 | $-6.1134 \cdot 10^{-4}$ | 0.9978 | |

| pH | $a_4$ | $b_4$ | $c_4$ | $d_2$ | $R^2$ |
|---|---|---|---|---|---|
| 3.00 | −17.4724 | 90.4444 | 7.7106 | 23.6154 | 0.9936 |
| 4.00 | −6.7752 | 84.1151 | 5.7375 | 17.8348 | 0.9983 |
| 5.00 | −8.4759 | 87.1013 | 5.0523 | 18.5511 | 0.9987 |
| 6.00 | −7.0781 | 88.2369 | 2.7482 | 18.9539 | 0.9979 |

| pH | $a_5$ | $b_5$ | $c_5$ | $R^2$ | |
|---|---|---|---|---|---|
| 3.00 | 8.2587 | −0.1556 | −0.4554 | 0.9965 | |
| 4.00 | 4.0043 | −0.0598 | −0.2987 | 0.9993 | |
| 5.00 | 4.6153 | −0.0753 | −0.3308 | 0.9995 | |
| 6.00 | 3.4923 | −0.0487 | −0.2758 | 0.9984 | |

in the two-phase equilibrium, respectively. Table 3 lists the fitting parameters and the estimated plait point values. The linear Eq. (6) provided a satisfactory fit for the studied system, especially at pH 3.00, as indicated by the correlation coefficients ($R^2$). Fig. 5 shows the locus of the estimated critical point for this system at pH = 5.00. In the ternary phase diagram, the slope of the tie-line changes
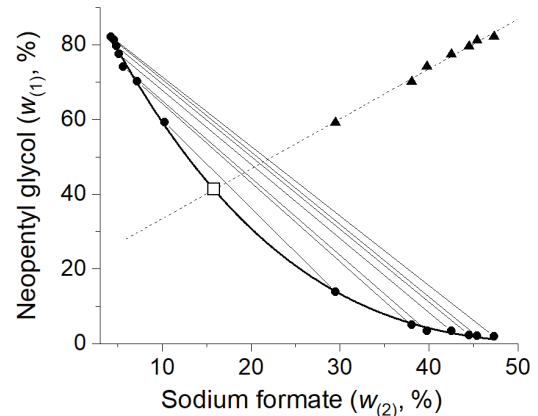


F i g .  5 – *Binodal curve, tie-lines and plait point for the neopentyl glycol + sodium formate + water system at pH=5.00, ((□), plait point)*

with component concentration. As the two-phase area diminished, the tie-line length also decreased until it reached the critical point, where the two liquid phases were in equilibrium with each other and formed a single liquid phase.

**Binodal curve correlation**

To further examine the liquid-liquid equilibrium mechanism in this system, the experimental data was analyzed using the least squares regression method. The data was fitted with polynomial (Eqs. (7) and (8)), exponential (Eqs. (9) and (10)) functions, and Pirdashti (Eq. (11)) model[22]. Several nonlinear equations took the following forms:

$$w_{(1)} = a_1 + b_1 w_{(2)} + c_1 w_{(2)}^2 + d_1 w_{(2)}^3 \tag{7}$$

$$w_{(1)} = a_2 - b_2 c_2^{w_{(2)}} \tag{8}$$

$$w_{(1)} = \exp\left(a_3 + b_3 w_{(2)} + c_3 w_{(2)}^2\right) \tag{9}$$

$$w_{(1)} = a_4 + b_4 \exp\left(\frac{c_4 - w_{(2)}}{d_2}\right) \tag{10}$$

$$w_{(1)} = \left(a_5 + b_5 w_{(2)}\right)^{\frac{-1}{c_5}} \tag{11}$$

The fitting parameters included $a_1$, $b_1$, $c_1$, $d_1$, $a_2$, $b_2$, $c_2$, $a_3$, $b_3$, $c_3$, $a_4$, $b_4$, $c_4$, $d_2$, $a_5$, $b_5$, $c_5$. The mass concentrations of neopentyl glycol and sodium formate are represented by $w_{(1)}$ and $w_{(2)}$, respectively. Fig. 2 shows the fitting curves at different pH values, while Table 4 lists the parameters and correlation coefficients $R^2$ obtained from the correlation of experimental equilibrium data. As shown in Fig. 2, the binodal curves exhibited a decreasing trend, while the two-phase area expanded with an increase in pH value. The high regression coefficient $R^2$ ($R^2 > 0.99$) obtained from the models used to correlate the equilibrium data and describe the binodal curves of the investigated system, confirms their re-

liability and satisfactory performance. Notably, the Pirdashti model and nonlinear empirical eqs. (7) and (9), yielded $R^2$ values very close to 1, indicating that the data predicted by these two equations are more closely aligned with the actual data. This suggests that the regression simulation is a more precise representation of the authentic experimental data and better describes the underlying trend.

## Conclusions

Liquid-liquid equilibrium data for the neopentyl glycol + sodium formate + water system were obtained at 303.15 K across pH values of 3.00, 4.00, 5.00, and 6.00. Phase diagrams for this ternary dissolution system were generated for each pH value. The impact of pH on the phase diagram was also investigated. The results indicated that increasing the pH slightly expanded the two-phase area. The systems' tie-line compositions were measured, revealing a direct relationship between pH increases, binodal curve expansion, and the length and slope of the tie-lines. Extrapolation was used to estimate the plait points' locations at various pH values, based on auxiliary curves. The experimental equilibrium data of this system were correlated using the Othmer-Tobias, Hand, and Bancroft equations. The consistency of the measured tie-line data is supported by the correlation coefficients, particularly with the Bancroft equation, which performed well in correlating the tie-line compositions, with correlation coefficient values $R^2$ ($R^2 > 0.99$). Additionally, the phase behavior of the system was successfully correlated using various polynomial, and exponential equations, and the Pirdashti model. The fitted curves closely matched the experimental data, and achieved fitting coefficient $R^2$ close to 1.

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